

Fast Analysis of Mn, Co, and Ni in the Ternary Cathode Material Mixture of Lithium Battery with EDXRF

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Abstract: The content of Mn, Co, and Ni in the ternary cathode material mixture of lithium batteries directly affects the electrochemical performance of the electrode material. The rapid measurement of the content of Mn, Co, and Ni is of great significance to the production and process control of the ternary cathode material. In this paper, energy dispersive X-ray fluorescence analysis (EDXRF) was used to analyze the content of Mn, Co, and Ni in the ternary cathode material mixture of lithium batteries. When calculating the peak area, the branch ratio subtraction method is used to correct the interference of the K_{β} peak of Co on the K_{α} peak of Ni. In the spectrum data processing, combining the advantages of the SNIP method and the polynomial fitting method to subtract the scattering background, the two methods are combined to process the measured spectrum lines, and good results have been achieved. In the matrix effect correction, by comparing the theoretical mass absorption coefficient and the experimental results, it is concluded that the Mn, Co, and Ni in the ternary cathode material mixture of the lithium battery are affected by the matrix effect, which is established by the multiple regression analysis in the empirical coefficient method. Mathematical model to correct the absorption enhancement effect of Mn, Co and Ni. The experimental results show that the average absolute error of Mn, Co, and Ni content in the ternary cathode material mixture of lithium battery using the empirical coefficient method is 0.09%, 0.02%, 0.04%, and the average relative error is 0.52%, 0.12%, 0.13%. Single sample analysis only needs 200s, which meets the requirements of fast, efficient and accurate analysis.

Keywords: EDXRF, Lithium Battery Ternary Cathode Material Mixture, Background Subtraction, Absorption and Enhancement Effect

1. Introduction

In order to cope with global climate change and protect the ecological environment, new energy industries such as solar energy, wind energy, and hydrogen energy have received strong policy support in my country. Since the 1990s, lithium batteries, which have the advantages of light weight, long life cycle, high energy density, etc., have been widely used in mobile phones, electric vehicles, power tools, digital cameras, aerospace and other industries, and are representative of modern high-performance batteries. It is also one of the power batteries with the best overall performance. In recent years, the lithium battery industry has also been developing rapidly, with its production capacity, output value, and

market share rising continuously. In 2014, China's lithium battery market share has become the world's first. Lithium batteries have four key main materials: positive electrode, negative electrode, electrolyte, and separator. Among them, the positive electrode material is an important part of the lithium battery, which directly determines the safety performance of the battery and whether the battery can be large-scale, accounting for about lithium battery cells. About 40% of material cost. The Mn, Co, and Ni elements in the ternary cathode material of lithium batteries influence each other and restrict each other. Changing their content ratio will produce materials with different characteristics. Therefore, accurate measurement of the content of Mn, Co, and Ni is important for the performance research of lithium batteries. Significant significance [1].

The main component analysis of lithium battery ternary cathode material mixture is mainly chemical analysis, mainly including EDTA titration and gravimetric method, ICP-AES method, ICP-MS method, ICP-OES method, etc. Among them, the EDTA titration and gravimetric method are cumbersome to operate and are not suitable for low-content impurity element testing; ICP-AES and ICP-MS are not suitable for simultaneous multi-element testing; ICP-OES is not suitable for low-content element testing. In recent years, the research on lithium battery ternary cathode materials has entered a stagnant state, but from the continuous expansion of the market audience, we can see that people's recognition of lithium batteries is relatively high [2].

Laboratory chemical analysis methods have low detection limits, high accuracy, and good accuracy, but they require longer time and complicated procedures, which cannot be completed on-site, so they cannot effectively guide production. XRF analysis has the advantages of low cost, portable instrument, fast analysis, and non-destructive analysis. It has been widely used in the fields of rapid on-site analysis, non-destructive testing and in-situ analysis, and online current-carrying analysis. It is also a broad analysis laboratory (test center). Standing analytical methods, especially on-site rapid analysis in the field of industrial production, are the most widely used analytical techniques [3-5]. In this paper, energy dispersive X-ray fluorescence analysis (EDXRF), which can quickly and accurately measure multiple elements at the same time, is used to analyze the content of Mn, Co, and Ni in the cathode material mixture of lithium batteries to provide accurate guidance for process control [6].

2. Principles and Methods

In 1913, British physicist Moseley discovered that the frequency ν of characteristic X-rays produced by high-energy electron bombardment of the target material has a certain functional relationship with the atomic number Z of the target material, and derives the Moseley law:

$$E_x = RhC(Z - \sigma)^2 \left(\frac{1}{n_f^2} - \frac{1}{n_i^2} \right) \quad (1)$$

In the formula, E_x represents the energy of characteristic X-ray fluorescence, R is Rydberg constant, 1096.776m^{-1} , h is Planck's constant, $6.626 \times 10^{-34} \text{ J}\cdot\text{s}$, C is the speed of light in vacuum, $3 \times 10^8 \text{ m}\cdot\text{s}^{-1}$, Z is the atomic number of the target material, σ is the shielding constant, usually positive, $\sigma=1$ in the K system, $\sigma=3.5$ in the L system, n_f and n_i are the main shells of the transition electron and the filling electron, respectively Quantum number [7, 8].

Moseley's law is the basic principle of X-ray fluorescence qualitative analysis. It can reflect that the characteristic X-ray energy of the element to be measured is proportional to the square of its atomic number Z . If the atomic number is different, the characteristic rays produced after being excited will also be different.

The purpose of the X-ray fluorescence quantitative analysis method is to calculate the content W_K of the element to be measured in the sample through the count rate I_K obtained by the measurement. Therefore, one can determine the material composition of the sample to be tested, calculate the proportional coefficient $K I_0 / (\mu_0 + \mu_K)$, and then calculate the content of the tested element W_K according to the counting rate I_K [7, 8].

$$I_K = K \cdot \frac{I_0}{\mu_0 + \mu_K} \cdot W_K \quad (2)$$

In the formula, μ_0 is the attenuation coefficient of incident radiation in the sample to be tested, and μ_K is the attenuation coefficient of the fluorescence spectrum to be tested in the sample to be tested.

It can be seen from formula (2) that the content of the element to be measured is proportional to its characteristic X-ray fluorescence intensity. In actual analysis, due to the mutual influence of the constituent elements of the test sample and the influence of other factors, the characteristic X-ray fluorescence intensity of the test element and its content are no longer a simple linear proportional relationship. But it is certain that the characteristic X-ray fluorescence intensity of the element to be measured has a great correlation with its content, and the quantitative analysis in X-ray fluorescence spectroscopy is based on this correlation.

3. Experimental Design

Malvern Panalytical Epsilon 1 desktop XRF spectrometer integrated machine, using Ag anode X-ray tube, the maximum voltage of X-ray tube is 50kV, the maximum current is 500 μ A, and the maximum power is 5W. The probe adopts SDD detector, the energy resolution (FWHM) of Mn-K $_{\alpha}$ is 135eV. Using USB and network to connect standard computer peripheral equipment, it can measure nearly one hundred elements between sodium (Na) and americium (Am) in different modes, and the detection limit is 1ppm-100%.

In this analysis, a batch of qualified ternary samples (C9) prepared by a lithium battery cathode material manufacturer in Chengdu was selected as the reference material, and 3 groups of different samples were artificially prepared according to the range of nickel, cobalt and manganese content in the samples, and the main matrix components of the 3 groups of samples were different. The changes are 18.18% manganese, 13.61% cobalt, and 30.91% nickel. In order to verify its matrix effect, the manganese content in the first group of samples was increased by 3.3%, 6.6%, and 10% respectively according to the gradient, and the cobalt content in the second group of samples was increased by 3.3%, 6.6%, and 10% respectively according to the gradient. The nickel content in the three groups of samples was increased by 1%, 4%, and 7% respectively according to the gradient, plus 1 original sample, for a total of 10 samples. The error of the powder sample is mainly caused by the unevenness of the sample. Therefore, the sample should be fully mixed in

multiple steps during sample preparation. In order to ensure the uniformity of the mixing, the powder sample is poured into a mortar for grinding and grinding. The time is more than 120 minutes [8]. The sample is prepared by the powder compaction method, the sample pressure of the compaction mechanism is 15MPa, and the pressure recovery time is 10~30s. The prepared samples are measured with a desktop XRF spectrometer for each element. The single measurement time for each sample is 200s, and the average value of multiple measurements is taken.

4. Results and Discussion

4.1. SNIP Method and Polynomial Fitting Method to Subtract Scattering Background

When using energy dispersive X-ray fluorescence method for qualitative analysis and quantitative analysis, spectral line smoothing and background subtraction are two key technologies, which will directly affect the accuracy of spectrum interpretation. This paper chooses the least square fitting method to smooth the full spectrum, and uses SNIP and polynomial fitting method to realize the scattering background.

Aiming at the problem that the iterative peak clipping method cannot automatically determine the appropriate number of iterations, Ryan proposed the SNIP (Sensitive Nonlinear iterative peak) algorithm in 1988. Generally, first use the LLS operator to transform the spectral line, the formula is as follows: The formulas should be clear and editable. They should be numbered as following:

$$y(n) = \ln(\ln(\sqrt{x(n)+1}+1)+1) \quad (3)$$

$$(n = 0, 1, \dots, N-1)$$

In the formula, $x(n)$ is the count of the multi-channel instrument spectrum in the n th channel, $y(n)$ is the transformed spectrum line of $x(n)$, and N is the total number of channels of the fluorometer. Starting from the step length $m=1$, increase the step length by one for each iteration, and stop the iteration when the step length is M .

Then perform the inverse transformation of the above formula to obtain the estimated background, the formula is:

$$x(n) = [\exp(e^{y(n)} - 1) - 1]^p - 1 \quad (4)$$

$$(n = 0, 1, \dots, N-1)$$

m is the maximum step length. When $2m+1$ is equal to the width of the peak base, the SNIP is optimal, and the background data is obtained.

The main background of the fluorescence spectrum obtained by the X-ray tube as the excitation source is the continuous X-ray spectrum, which is a relatively slow-changing curve. The polynomial fitting method aims to first perform polynomial fitting on the background of the background area, use the least square method to obtain the polynomial parameters, and then interpolate to obtain the

background of the peak area. The judgment basis selects 2 times the standard deviation of the background. When looking for the best fit times, search from small to large. First-degree polynomials and second-degree polynomials are prone to underfitting, so start searching from the third degree. Experiments have found that a polynomial of degree 30 can fit most backgrounds, and the maximum degree is 50. The spectral smoothing and background subtraction are realized by programming on the computer, and the calculation process is realized by MATLAB language [9-12].

In this paper, taking the background subtraction of characteristic peaks of nickel, cobalt and manganese as an example, we intercepted the 600 to 1100 smooth spectral lines of the measured sample. The background subtraction effect achieved by the SNIP and polynomial fitting method is shown in Figure 1, and the partial enlarged view is shown in Figures 1 and 2. It can be seen that the background subtraction effect of the characteristic peaks of nickel, cobalt and manganese affected by the background is very good, indicating that the SNIP method and the polynomial fitting method can effectively subtract the background and maintain the peak shape of each spectral peak [10].

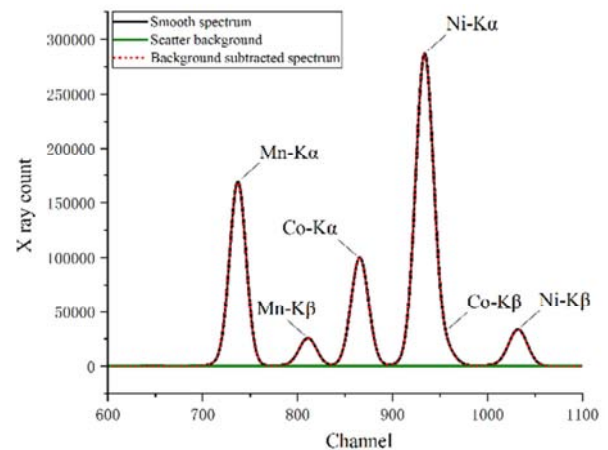


Figure 1. Schematic diagram of the background deduction effect of SNIP and polynomial fitting method on the spectrum.

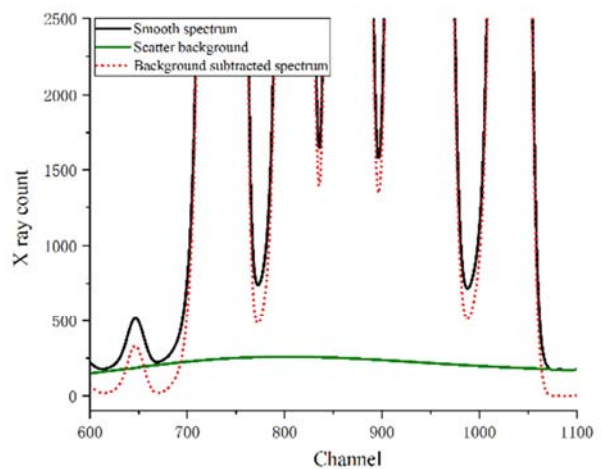


Figure 2. SNIP and polynomial fitting method on the background deduction effect of the spectrum partial enlarged view.

4.2. Decomposition of Overlapping Peaks

According to Moseley's law, when the atomic number is small, the energy difference between K_α and K_β of characteristic X-rays is very small. In the actual measurement process, due to the limitation of the energy resolution of the detector, the X-ray characteristic peaks overlap, and the mutual interference between the element X-ray characteristic peaks is an important reason that affects the measurement accuracy.

Table 1. Element K series characteristic X-ray.

Element	$K_{\alpha 1}(\text{keV})$	$K_{\alpha 2}(\text{keV})$	$K_{\beta 1}(\text{keV})$	$K_{\beta 2}(\text{keV})$
Mn	5.898	5.887	/	/
Co	6.930	6.915	7.649	/
Ni	7.477	7.460	8.264	8.328

It can be seen from Table 1 that the energy difference between the characteristic rays of Mn, Co and Ni is very small. When the characteristic X-ray energies produced by two elements are very close, for example, Co- K_β and Ni- K_α . For the above two characteristic X-ray, due to the energy resolution of the detector, the two characteristic X-ray peaks cannot be completely separated, forming a combined peak. When calculating the peak area, mutual interference occurs, which affects the accuracy of fluorescence analysis [8]. The simplest and the earliest practiced method for decomposing overlapping peaks is the branch ratio subtraction method, which corrects the count rate based on the proportional relationship between the spectral peaks. There are still many new methods, such as the introduction of mathematical methods such as neural networks, wavelets, etc., to promote the development of spectral understanding technology.

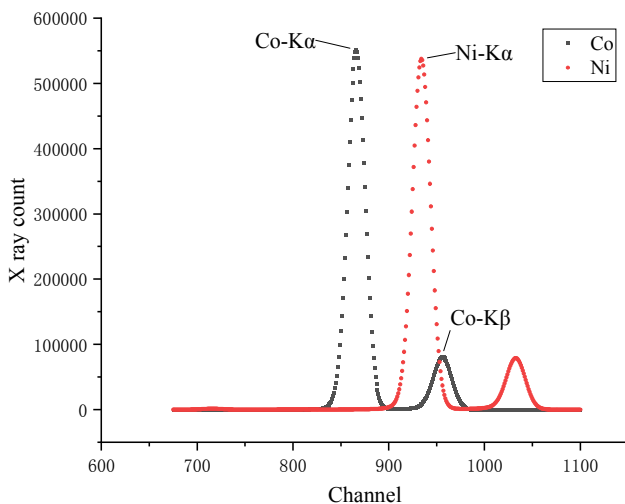


Figure 3. Spectra of pure cobalt and pure nickel.

Figure 3 shows the spectrum of pure cobalt and pure nickel. In the figure, it can be clearly seen that the Co- K_α

line is not affected by the elements before and after, but the Ni- K_α line is interfered by the Co- K_β line. In this paper, the branch ratio subtraction method is used to correct the interference of the K_β of the previous element to the K_α peak of the latter element. The specific method is as follows:

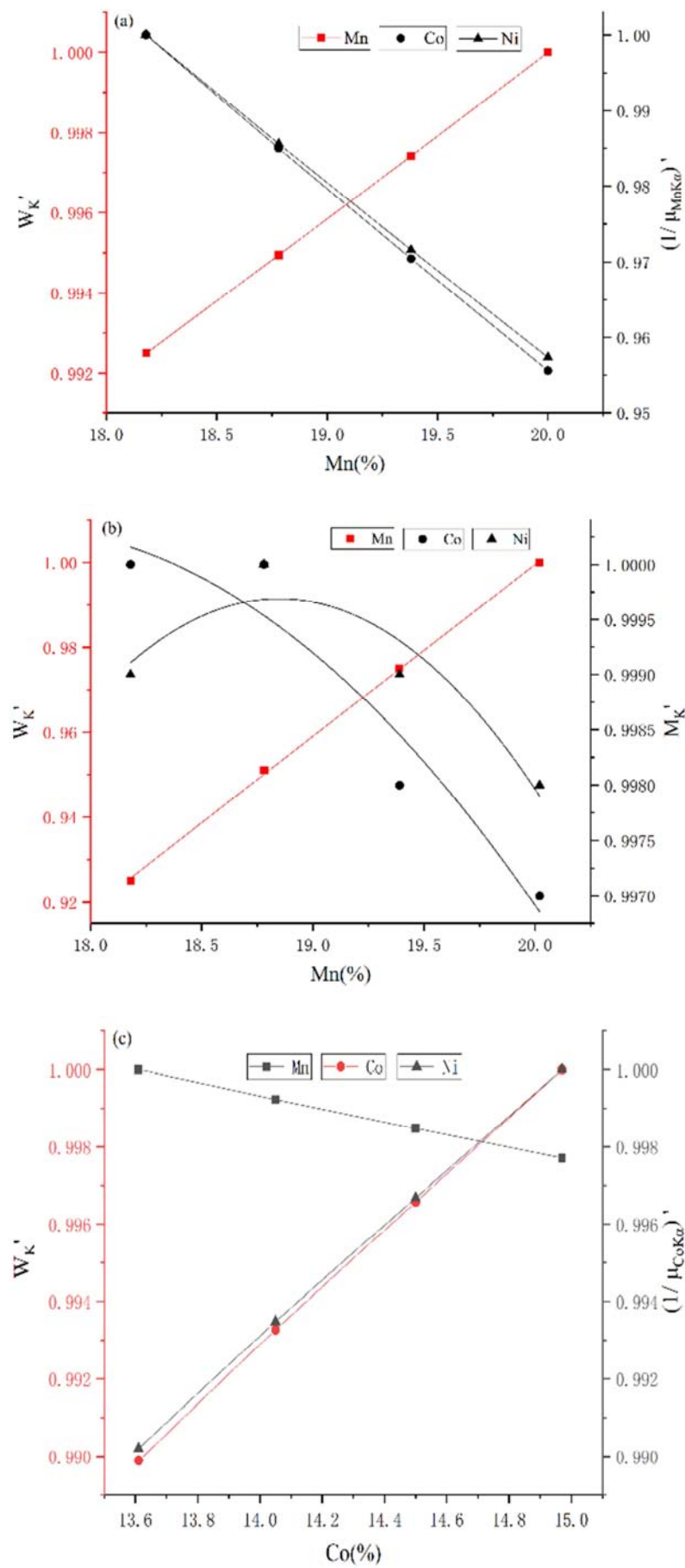
$$I_{Ni} = I_{Ni}' - K_b I_{Co} \quad (5)$$

In the formula, I_{Ni} and I_{Co} are the K_α peak intensities of nickel and cobalt, I_{Ni}' is the K_α measurement channel of the instrument nickel, and K_b is the branching ratio of cobalt element K_α and K_β (can be measured by experiment).

4.3. Absorption Enhancement Effect and Correction Among Mn, Co, Ni

The absorption enhancement effect between elements is generally caused by the following two situations: general absorption effect and special absorption effect. The general absorption effect is the change of the mass absorption coefficient μ caused by the change of the matrix composition in the sample. The special absorption effect is generated when the characteristic X-ray energy of the analysis element and the matrix element is higher than and close to the absorption limit. If the absorption limit of the analysis element is slightly lower and the characteristic X-ray energy of the matrix element, the excitation efficiency of the analysis element will be increased, if on the contrary, the characteristic spectrum of the analysis element will be absorbed by the matrix element and reduced [8, 12].

It can be seen from formula (2) that the proportional coefficient $KI_0/(\mu_0 + \mu_K)$ in the sample is proportional to the ratio (M_K) of the counting rate I_K to the content of the element to be measured W_K . When it is determined that the relative positions of the element to be measured, the detector and the excitation detection device remain unchanged, K is a constant. This experiment uses X-ray tube excitation, and I_0 is the primary radiation exposure rate of the excitation source, which can also be regarded as a constant [7]. It can be seen that the change of the combined mass absorption coefficient in the mixed sample will cause a non-linear change between the element count rate and the content. In the actual sample analysis, the content of each element is uncertain, and there is no way to know the value of the combined mass absorption coefficient. And there is no way to know the extent of the influence of the combined mass absorption coefficient on the relationship between M_K' - W_K (M_K' is the normalized value). Therefore, it is necessary to conduct an experimental analysis. When the element content in the mixed sample is different, study the absorption enhancement effect on the count rate. The degree of influence of the rate [12].



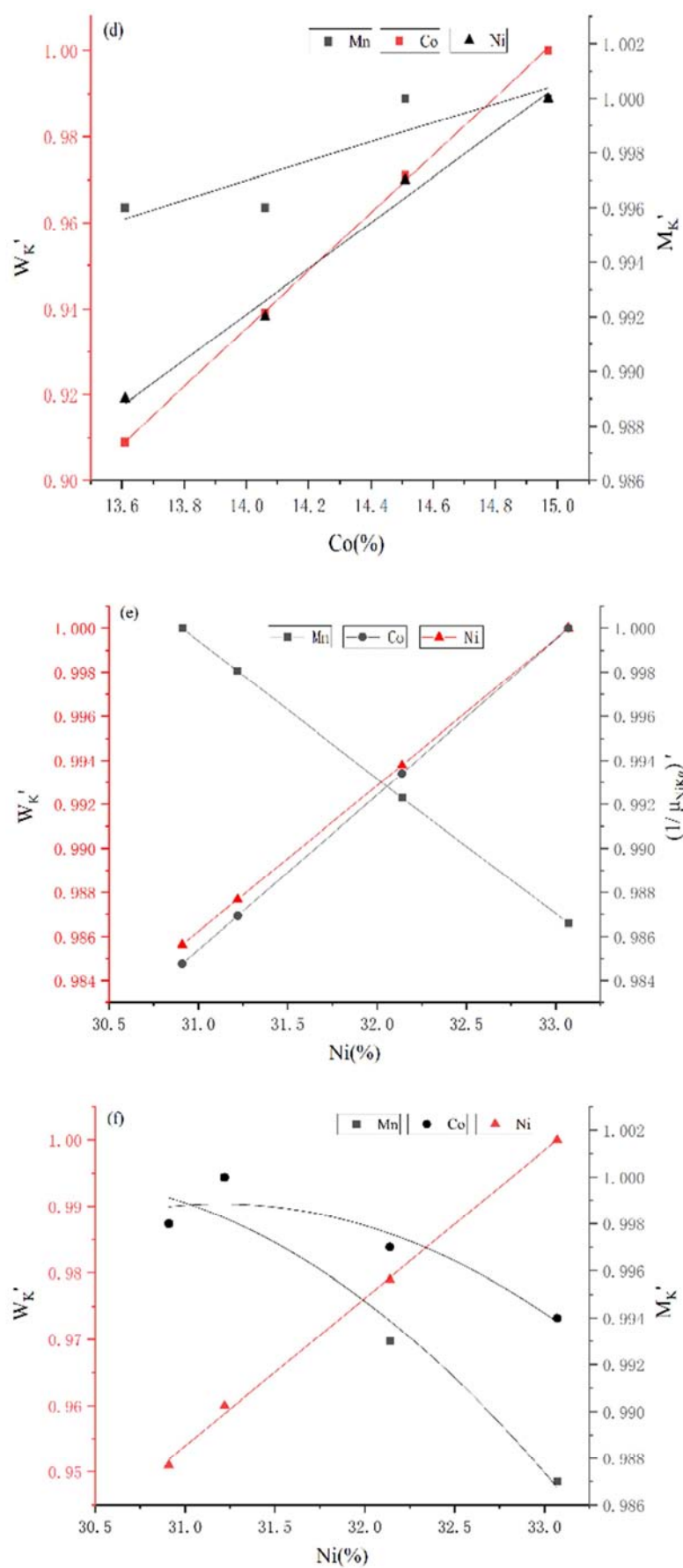


Figure 4. The relationship between the combined mass absorption coefficient and content of nickel, cobalt and manganese in the sample.

It can be seen from Figure 4 that the ratio (M_K) of the measured element content W_K to the counting rate I_K in the actual measurement does not show a linear relationship like the theoretical calculation result. This is caused by the absorption enhancement effect between elements and needs to be corrected. In fact, the elements that have a significant contribution to the matrix effect are those elements that have high content, large content changes, and strong absorption or enhancement of the characteristic fluorescence to be measured. Therefore, by processing such major interference elements separately, a more accurate matrix effect correction effect will be obtained. At this time, the mathematical correction method can be used, which is characterized by quantifying the interaction between the elements in the matrix and expressing it in various mathematical models, so that various calculation

methods can be used to complete the correction of the matrix effect, mainly the correction of the effect between the elements [3-5, 13]. Matrix effect correction adopts multivariate statistical analysis of empirical coefficient method, and the established mathematical model is as follows:

$$W_i = a_{i0} + a_i I_i + \sum_{j=0, j \neq i}^N a_{ij} I_j \quad (6)$$

In the formula, W_i is the content of the element to be measured, I_i and I_j are the intensities of the element to be measured and the interference element, respectively; a_{i0} , a_i , and a_{ij} are empirical coefficients, which are obtained through multiple linear regression analysis.

Table 2. Analysis results of Mn in the mixture of ternary cathode materials for lithium batteries.

Serial number	Mn (%)			
	Actual value	Analysis value	Absolute error	Relative error
C9-0	18.18	18.26	0.07	0.41
C9-1	18.78	18.81	0.03	0.13
C9-2	19.39	19.35	-0.04	-0.19
C9-3	20.02	19.89	-0.12	-0.61
C9-4	18.04	18.10	0.06	0.32
C9-5	17.91	18.02	0.11	0.63
C9-6	17.77	17.87	0.10	0.58
C9-7	18.07	18.16	0.10	0.54
C9-8	17.71	17.63	-0.08	-0.46
C9-9	17.36	17.13	-0.23	-1.33
Average value	/	/	0.09	0.52

Table 3. Analysis results of Co in the mixture of ternary cathode materials for lithium batteries.

Serial number	Co (%)			
	Actual value	Analysis value	Absolute error	Relative error
C9-0	13.61	13.62	0.01	0.09
C9-1	13.42	13.44	0.01	0.09
C9-2	13.24	13.23	-0.01	-0.05
C9-3	13.05	13.04	-0.02	-0.12
C9-4	14.06	14.05	-0.01	-0.08
C9-5	14.51	14.52	0.01	0.09
C9-6	14.97	14.94	-0.02	-0.14
C9-7	13.52	13.55	0.04	0.26
C9-8	13.25	13.26	0.01	0.05
C9-9	12.99	12.97	-0.02	-0.19
Average value	/	/	0.02	0.12

Table 4. Analysis results of Ni in the mixture of ternary cathode materials for lithium batteries.

Serial number	Ni (%)			
	Actual value	Analysis value	Actual value	Relative error
C9-0	30.91	C9-0	30.91	C9-0
C9-1	30.50	C9-1	30.50	C9-1
C9-2	30.08	C9-2	30.08	C9-2
C9-3	29.65	C9-3	29.65	C9-3
C9-4	30.67	C9-4	30.67	C9-4
C9-5	30.45	C9-5	30.45	C9-5
C9-6	30.21	C9-6	30.21	C9-6
C9-7	31.22	C9-7	31.22	C9-7
C9-8	32.14	C9-8	32.14	C9-8
C9-9	33.07	C9-9	33.07	C9-9
Average value	/	/	0.04	0.13

The empirical coefficient method was used to analyze the content of Mn, Co, and Ni in the ternary cathode material mixture samples of lithium batteries. The analysis results are shown in Tables 2 to 4. The unitary linear regression analysis was performed on the Mn element, and the correlation $r^2=0.981$ was obtained. The average absolute error of the Mn content analysis result was 0.09%, and the average relative error was 0.52%; for the Co element, the binary linear regression analysis was performed to obtain the correlation. $r^2=0.999$, the average absolute error of the Co content analysis result is 0.02%, the average relative error is 0.12%; the binary linear regression analysis for Ni element, the correlation $r^2=0.998$, the average absolute error of the Ni content analysis result is 0.04%, the average relative error is 0.13%. Therefore, calibration models for analyzing Mn, Co, and Ni in mixed samples of lithium battery ternary cathode materials can be established respectively.

$$W_{Mn} = -2.076 + 5.495 \times 10^{-6} I_{Mn} \quad (7)$$

$$W_{Co} = 0.366 + 5.780 \times 10^{-6} I_{Co} - 1.398 \times 10^{-8} I_{Mn} \quad (8)$$

$$W_{Ni} = -1.648 + 5.102 \times 10^{-6} I_{Ni} - 1.595 \times 10^{-6} I_{Co} \quad (9)$$

5. Conclusion

It can be seen from the experimental data that the EDXRF method is fast in the determination of Mn, Co, and Ni in the lithium battery ternary cathode material mixture, and the data is true and effective. The empirical coefficient method is used to correct the matrix effect, and the correction effect is better. It can be used for rapid detection of element content in lithium battery ternary cathode material mixture in industrial production. The detection speed is fast, efficient, convenient, and has good economic benefits. However, current methods alone are not enough to realize the high-precision and rapid detection analysis required in industrial production. Therefore, in order to further improve the accuracy of the EDXRF method for determining the Mn, Co, and Ni in the ternary cathode material mixture of lithium batteries, the following methods can be tried when processing the spectrum data: (1) Study the changes of the energy spectrum background of different types of samples Rules, especially the influence of the background of the characteristic peak integration interval of each element to be measured with the change of the element content ratio, establish an accurate background subtraction method and its mathematical model. (2) During the high-speed detection and analysis of samples, they are often susceptible to insufficient energy resolution of the X-ray fluorescence detector. The characteristic X-ray energy peaks of each element actually measured and recorded by the spectrometer overlap or are close to each other. This makes it impossible to accurately determine the characteristic X-ray peak positions of each element. It is necessary to study sophisticated spectral decomposition techniques, especially the overlapping decomposition

techniques between Co- K_β and Ni- K_α , and establish accurate spectral decomposition mathematical models. In addition, the factors that make qualitative analysis difficult are the existence of sum peak effects, escape peaks and various scattered scattering peaks. These problems also require follow-up experiments to explore the laws and lay the foundation for high-precision X-ray fluorescence analysis.

This test lays a solid foundation for the analysis of the high-precision and rapid detection of the lithium battery ternary cathode material mixture in the future. At the same time, linear multivariate fitting needs to be perfected in the quantitative analysis of Mn, Co, and Ni in the mixture of lithium battery ternary cathode materials. The matrix correction in the Mn, Co, and Ni in the mixture is improved.

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